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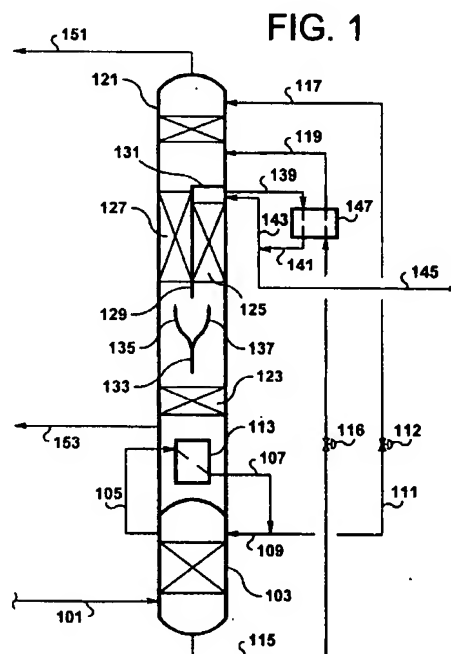
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(54) **Process for distillation of multicomponent fluid suitable for production of an argon-enriched stream from a cryogenic air separation process**

(57) A multicomponent feed (101) is separated by distillation into at least one stream (151) enriched in the most volatile component, one stream (153) enriched in the least volatile component, and one stream (145) enriched in a component of intermediate volatility using a distillation system comprising a distillation column (121) having a partitioned section (125) adjacent an intermediate distillation section (127) and closed at one end (131). The geometry of the partitioned section (125) minimizes vapour and liquid maldistribution by having an equivalent diameter ( $D_e$ ) at least 60% of the ideal diameter ( $D_i$ ) of the partitioned section. The process has particular application to the production of an argon-enriched stream by cryogenic air separation.



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## Description

[0001] The present invention relates to the separation of a multicomponent feed by distillation into at least three streams: at least one enriched in a most volatile component, at least one enriched in a least volatile component, and at least one enriched in a component of an intermediate volatility. The separation is carried out using a distillation column having a partitioned section to recover a component of intermediate volatility. The present invention also relates to the production of an argon-enriched stream from a cryogenic air separation process using a partitioned section within a primary distillation column to rectify and enrich an argon-bearing stream.

[0002] The traditional method of recovering argon from air is to use a double-column distillation system having a higher pressure column and a lower pressure column thermally linked with a reboiler/condenser and a side-arm rectifier column attached to the lower pressure column. Oxygen product is withdrawn from the bottom of the lower pressure column and at least one nitrogen-enriched stream is withdrawn from the top of the lower pressure column. Vapour provided by the reboiler of the lower pressure column rises through the bottom section of that column then splits into two portions. A first portion continues up the lower pressure column into an intermediate distillation section above. A second portion is withdrawn from the lower pressure column and passed to the side-arm column. This portion, which generally contains between 5% and 15% argon, traces of nitrogen, and the balance oxygen, is rectified in the side-arm column to produce an argon-enriched stream substantially purified of oxygen. Typically, this argon-enriched stream, commonly referred to as crude argon, is withdrawn from the top of the side-arm column with an oxygen content ranging from parts per million (ppm) levels to 3 mole%. The rectification in the side-arm column is achieved by providing liquid reflux via a condenser located at the top of the side-arm column.

[0003] Since vapour is withdrawn from the lower pressure column to feed the side-arm column, the vapour flow to the intermediate section of the lower pressure column is necessarily reduced relative to the vapour flow in the bottom section of the lower pressure column. Commonly, steps must be taken to maintain proper mass transfer performance in the intermediate section, such as reducing the diameter of the column in the intermediate section to maintain appropriate vapour velocity and/or reducing the packing density to maintain appropriate liquid loading.

[0004] In general, whenever a side rectifier or a side-stripper is employed, vapour and liquid flow rates in the intermediate distillation section of the main column (e.g., a lower pressure column) are reduced relative to the flow rates in the distillation section below and/or the distillation section above.

[0005] Divided-wall columns have been proposed in

the literature as a means to better utilize a given column diameter, and thereby reduce capital cost. Divided-wall columns essentially contain multiple distillation sections at the same elevation within a single column shell. An early example of the use of a divided-wall column is disclosed in US-A-2,471,134 (Wright). Wright shows how a partitioning wall may be used to produce three products from a single distillation column. In Wright, the partition forms a separation zone, the top and bottom of which communicates with the main distillation column. Divided-wall columns of the type disclosed by Wright are discussed further by Lestak and Collins in "Advanced Distillation Saves Energy and Capital", Chemical Engineering, pages 72-76, July 1997. Christiansen, Skogestad, and Lien disclose further applications for divided-wall columns in "Partitioned Petlyuk Arrangements for Quaternary Separations", *Distillation and Absorption '97*, Institution of Chemical Engineers, Symposium Series No. 142, pages 745-756, 1997.

[0006] In "Multicomponent Distillation - Theory and Practice", by Petlyuk and Cerfimow (page 198, figure VI-4e, published by Moscow Chemie, 1983) the authors disclose a configuration for a divided-wall column where the partitioning wall is cylindrical and forms an annular separation zone, the top and bottom of which communicates with the main distillation column.

[0007] US-A-5,946,942 (Wong, et al.) discloses an application of divided-wall principles to air separation. Wong discloses an apparatus wherein the lower pressure column contains an inner annular wall. The region contained between the inner annular wall and the outer shell of the lower pressure column constitutes a section for the production of argon product. A drawback of this divided-wall column for argon recovery stems from the geometry of the device used, as explained below.

[0008] The cross-sectional geometry of the argon rectification section taught by Wong is annular. At the top of the annular section, the rising vapour must be collected and withdrawn. If a single outlet pipe is used, vapour from the farthest location in the annulus must travel significantly farther than vapour from the nearest location. This introduces flow maldistribution of vapour within the separation section below. Similarly, maldistribution of liquid also is a concern, especially if the separation section below uses packing. It is possible to mitigate maldistribution by taking steps, such as using multiple outlet and inlet pipes, but the result is a more complex and costly design. Furthermore, use of an annular geometry produces a relatively large wall surface area. Large wall surface area is discouraged when packing is used, because liquid tends to migrate to the walls, thereby introducing liquid flow maldistribution.

[0009] It is desired to have a process using the divided-wall concept which minimizes vapour and liquid maldistribution in the argon section of a distillation column.

[0010] It is further desired to have a process using the divided-wall concept which minimizes vapour and liquid

maldistribution in any partitioned section used to recover a component enriched in an intermediate-volatility component.

**[0011]** It also is desired to have a process for separation of a multicomponent fluid which overcomes the difficulties and disadvantages of the prior art to provide better and more advantageous results.

**[0012]** The present invention provides a process for distillation of a multicomponent fluid containing at least three components, each component having a different volatility, into at least three streams.

**[0013]** The process uses a distillation column system comprising at least a first distillation column having at least a first distillation section, a second distillation section, an intermediate distillation section between said first and second distillation sections and a partitioned section adjacent the intermediate distillation section. A vertical separating element and an adjacent end separating element isolate the partitioned section from the intermediate distillation section. The equivalent diameter ( $D_e$ ) of the partitioned section is at least 60% of the ideal diameter ( $D_i$ ) of the partitioned section. The multicomponent fluid is fed to the distillation column system, wherein a first portion of a fluid stream flows into the intermediate distillation section and a second portion of the fluid stream flows into the partitioned section. A side stream enriched in a component having an intermediate is withdrawn from the partitioned section.

**[0014]** The equivalent diameter ( $D_e$ ) is four times the cross-sectional flow area enclosed by the vertical separating element divided by the perimeter formed by the vertical separating element, and the ideal diameter ( $D_i$ ) is the diameter of a circle which has the same cross-sectional flow area as that cross-sectional flow area which is enclosed by the vertical separating element.

**[0015]** The present invention further provides a distillation column having a first distillation section and a second distillation section; an intermediate distillation section between the first distillation section and the second distillation section; and a partitioned section adjacent the intermediate distillation section. The partitioned section has a vertical separating element and an end separating element adjacent the vertical separating element, wherein the vertical and end separating elements isolate the partitioned section from the intermediate distillation section, and the equivalent diameter ( $D_e$ ) of the partitioned section is at least 60% of the ideal diameter ( $D_i$ ) of the partitioned section.

**[0016]** A stream enriched in a component having the highest volatility can be withdrawn from the location above at least one distillation section above the intermediate distillation section and a stream enriched in a component having the lowest volatility can be withdrawn from a location below at least one distillation section below the intermediate distillation section.

**[0017]** The fluid stream can be a vapour rising from a distillation section and a liquid can be fed to the partitioned section at a location adjacent the top of the par-

tioned section. Said liquid can be produced by at least partially condensing at least a portion of a vapour leaving the partitioned section.

**[0018]** Alternatively, the fluid stream can be a liquid descending from a distillation section above the intermediate distillation section and a vapour can be fed to the partitioned section at a location adjacent the bottom of the partitioned section. Said vapour can be produced by at least partially vaporizing a portion of the liquid leaving the partitioned section.

**[0019]** The vertical separating element can be cylindrical or a vertical wall attached to a cylindrical wall of the first distillation column.

**[0020]** The side stream can be transferred to at least one other distillation column.

**[0021]** The present invention is applicable to the distillation of various multicomponent fluids containing at least three components. For example, the multicomponent fluid may be selected from benzene/toluene/xylene mixtures, nitrogen/carbon monoxide/methane mixtures, combinations of three or more components from  $C_1$  to  $C_5$  alcohols, and hydrocarbon mixtures, said hydrocarbon mixtures being selected from pentane-hexane-heptane, isopentane-pentane-hexane, butane-isopentane-pentane, iso-butane-n-butane-gasoline, and combinations of three or more components from  $C_1$  to  $C_6$  hydrocarbons or  $C_4$  isomers.

**[0022]** As another example, the multicomponent fluid may be a nitrogen/oxygen/argon mixture, especially air, and the at least three components are nitrogen having a highest volatility, oxygen having a lowest volatility, and argon having an intermediate volatility between the highest volatility and the lowest volatility.

**[0023]** Preferably, the argon-enriched stream withdrawn from the partitioned section has an oxygen content of less than 60 mole %.

**[0024]** The argon-enriched stream can be transferred to at least one other distillation column or to an adsorption separation system.

**[0025]** The present invention also provides a cryogenic air separation unit using a process of the invention.

**[0026]** The present invention provides a process for the separation of a multicomponent feed in a distillation system having at least one distillation column that produces at least one stream enriched in the most volatile component from the top of the column, at least one stream enriched in the least volatile component from the bottom of the column, and at least one stream enriched in a component of intermediate volatility from a partitioned section within the column. In the process:

- a) a fluid stream from within the at least one distillation column is split into at least two portions;
- b) a first portion from step a) flows into an intermediate distillation section of the at least one distillation column;
- c) a second portion from step a) flows into a partitioned section of the at least one distillation column,

said partitioned section comprising a vertical separating element and an end separating element to isolate said partitioned section from the intermediate distillation section at all locations except at the inlet of said partitioned section;

d) said second portion flows through the partitioned section and is removed from said partitioned section as a stream enriched in a component of intermediate volatility;

e) the equivalent diameter of the partitioned section is at least 60% of the ideal diameter of the partitioned section.

**[0027]** The process has particular application to the cryogenic separation of air to produce at least a nitrogen-enriched stream from the top of the column, an oxygen product stream from the bottom of the column, and an argon-enriched stream from a partitioned section within the column.

**[0028]** The following is a description by way of example only and with reference to the accompanying drawings of presently preferred embodiments of the invention. In the drawings:

Figure 1 is a schematic diagram of an embodiment of the present invention;

Figure 2A is a schematic isometric view of a partitioned section in a distillation column used in the present invention;

Figure 2B is a schematic top view of a partitioned section in a column used in the present invention;

Figure 3 illustrates various top views of different types of partitioned section geometries;

Figure 4 illustrates various top views of additional types of partitioned section geometries;

Figure 5 is a schematic diagram of another embodiment of the present invention;

Figure 6 is a schematic diagram of another embodiment of the present invention;

Figure 7 is a schematic diagram of an embodiment of the present invention for separation of a four-component mixture; and

Figure 8 is a schematic diagram of another embodiment of the present invention for the separation of a four-component mixture.

**[0029]** To illustrate the concept of equivalent diameter and further describe the present invention, an example based on the separation of air is shown in Figure 1 (which illustrates the process), and Figures 2A and 2B illustrate the geometry of the partitioned section. For the purpose of illustration, the multicomponent feed comprises nitrogen, the most volatile component, oxygen, the least volatile component, and argon, the component of intermediate volatility.

**[0030]** In Figure 1, a compressed feed air stream, free of heavy components (such as water and carbon dioxide) and cooled to a suitable temperature, is introduced

as stream 101 to the bottom of a higher pressure column 103. The pressure of this feed air stream generally is greater than 3.5 atmospheres (0.35 MPa) and less than 24 atmospheres (2.4 MPa), with a preferred range of 5 to 10 atmospheres (0.5 to 1 MPa). The feed to the higher pressure column is distilled into a higher pressure nitrogen vapour stream 105 at the top of a column and a crude liquid oxygen stream 115 at the bottom of the column. Nitrogen vapour stream 105 is condensed in reboiler/condenser 113 to produce liquid stream 107, which subsequently is split into two streams, 109 and 111. Stream 109 is returned to the higher pressure column as reflux; stream 111 is reduced in pressure by valve 112 and is directed to a top of the lower pressure column 121 as reflux stream 117. Although not shown (for simplicity), lower pressure column reflux stream 111/117 often is cooled via indirect heat exchange with another stream prior to introduction to lower pressure column 121. Crude liquid oxygen stream 115 is subjected to optional indirect heat exchange(s), reduced in pressure by valve 116, and introduced to the lower pressure column as stream 119.

**[0031]** The feeds to the lower pressure column 121 are distilled into a lower pressure nitrogen vapour stream 151 at the top of the column and an oxygen stream 153 at the bottom of the column. Vapour stream 133 exits bottom distillation section 123 of the lower pressure column and may contain between 3% to 25% argon but typically contains between 5% to 15% argon. Stream 133 is split into two fractions: a first portion 135 and a second portion 137.

**[0032]** The first portion 135 flows into an intermediate distillation section 127. The second portion 137 flows into a partitioned section 125, which comprises a vertical separating element 129 and an end separating element 131 to isolate the partitioned section 125 from the intermediate distillation section 127. Vapour stream 137 rises through the partitioned section 125 and is rectified to produce argon-enriched stream 139. Stream 139 is at least partially condensed in heat exchanger 147 to produce stream 141, which subsequently is split into two streams, 143 and 145. Stream 143 is returned to the partitioned section 125 as reflux; stream 145 is removed from the distillation system. The refrigeration for heat exchanger 147 is provided by partially vaporizing the crude liquid oxygen stream 115 after it has been reduced in pressure through valve 116.

**[0033]** Ideally, intermediate section 127 requires approximately 20 to 25 stages of separation. If the partitioned section 125 comprises a similar number of separation stages, then the oxygen content in the argon-enriched stream is nominally 10 mole% but may range between 3 mole% and 60 mole%. The purity of this argon-enriched stream is sufficient to be rejected from the distillation system without significantly increasing the loss of oxygen. In fact, operation in such an "argon-rejection" mode will increase the oxygen recovery of the distillation system, since inclusion of the partitioned col-

umn makes the oxygen-argon separation easier in bottom distillation section 123 of the lower pressure column.

**[0034]** Figures 2A and 2B show one possible configuration for the partitioned section. Referring to Figure 2A, the vertical separating element 129 comprises a vertical plate and that portion of the column wall which is in contact with partitioned section 125. When viewed from the top (Figure 2B), the vertical plate forms a line of length L and that portion of the column wall in contact with the partitioned section forms an arc of length C. The end separating element 131, when viewed from the top (Figure 2B) has an area A. Argon-enriched vapour stream 139 and partitioned section reflux stream 143 (to the partitioned section) are shown as leaving/entering the column via vapour outlet pipe 138 and conduit 142. Alternatively, these streams may enter/leave through the end separating element 131.

**[0035]** The cross-sectional flow area enclosed by the vertical separating element is shown as the shaded region in Figure 2B and is denoted as A. The perimeter formed by the vertical separating element is the projected length L of the vertical plate plus the projected length C along the column wall. The equivalent diameter ( $D_e$ ) is a term commonly used in fluid flow and is four (4) times the cross-sectional flow area divided by perimeter. For this example:

$$D_e = 4A/(L+C)$$

The equivalent diameter provides a measure of "roundness". Ideally, it would be desired for the top view (Figure 2B) of the partitioned section to be circular. This geometry would tend to make the vapour flow path from the various positions in the partitioned section to the outlet nozzle (vapour outlet pipe 138) more uniform and thereby reduce vapour flow maldistribution. In addition, a circular geometry has the minimum perimeter and is most appropriate for minimizing the flow of liquid down the wall. If the top view (Figure 2B) of the partitioned section is circular and the area of the projection is A, then the ideal diameter ( $D_i$ ) would be:

$$D_i = (4A/\pi)^{1/2}$$

In accordance with the present invention, the ratio of the equivalent diameter to the ideal diameter ( $D_e/D_i$ ) must be greater than 0.6.

**[0036]** The cross-sectional area of the vapour outlet pipe 138 for argon-enriched stream 139 is typically 10% of the cross-sectional area A. Note that, for clarity, the vapour outlet pipe shown in Figure 2 is disproportionately small.

**[0037]** Figures 3 and 4 illustrate top views of different types of partitioned section geometries. Figures 3 and 4 are drawn to scale with the cross-sectional area of the

outlet pipe, shown as a solid black circle, equal to 10% of the cross-sectional area of the partitioned section, shown as a cross-hatched region. Figure 3 corresponds to a partitioned section having a cross-sectional area that is 25% of the total column cross-sectional area. Figure 4 corresponds to a partitioned section having a cross-sectional area that is 50% of the total column cross-sectional area. Cross-sectional areas required to practice the present invention typically lie within the range shown by Figures 3 and 4.

**[0038]** Figure 3(a) shows a cylindrical partitioned section located in the centre of the column cross-section. Figure 3(b) shows the same cylindrical partitioned section at a location offset from the centre of the column. Figure 3(c) shows a partitioned section which is bounded between a chord and the column wall. Figures 3(d), 3(e), and 3(f) show a sector, an equilateral triangle, and a square, respectively. Figure 3(g) shows a partitioned section bounded by two chords. Finally, Figure 3(h) shows the prior art configuration taught by US-A-5,946,942 (Wong et al.).

**[0039]** The ratio of the equivalent diameter to the ideal diameter ( $D_e/D_i$ ) also is shown for each configuration. For the configurations represented by Figures 3(a) through Figure 3(f), most regions of the cross-hatched area are within one pipe diameter of the nozzle (vapour outlet pipe). In Figure 3(g), most regions of the cross-hatched area are within two pipe diameters of the nozzle (vapour outlet pipe). For the prior art configuration taught in Figure 3(h), two features can be seen. First, it is not possible to project the nozzle onto the cross-sectional area. Second, the path lengths that must be travelled by vapour elements leaving the cross-hatched areas vary substantially.

**[0040]** As previously indicated, Figure 3 reflects a relatively small partitioned section (i.e., 25% of the total column cross-sectional area). Figure 4 shows the same configurations for the case when the partitioned section occupies 50% of the total column cross-sectional area. Here, Figures 4(a) through 4(g) illustrate that most regions of the cross-hatched area are within two pipe diameters of the nozzle (vapour outlet pipe). Again, the prior art configuration in Figure 4(h) is subject to the same limitations that were discussed for Figure 3(h).

**[0041]** Of the configurations shown in Figures 3(a) through 3(g) and Figures 4(a) through 4(g), some illustrate some portion of the partitioned section in contact with the outer wall of the main distillation column. This allows intermediate feeds to be introduced to and/or intermediate products to be withdrawn from the intermediate distillation section without penetrating or passing through the partitioned section.

**[0042]** In discussing the embodiment of Figure 1 it was noted that the oxygen content of argon-enriched stream 139 can be fairly substantial and may not be suitable for delivery to the customer. The purity of the argon-enriched stream may be increased by extending the partitioned section upwards in the column beyond the loca-

tion where intermediate section 127 ends. This adds stages of separation to the partitioned section and permits the production of a higher purity argon stream. It may be desirable in certain cases to extend the partitioned section all the way to the top of the distillation column 121, whereby the end separating element may be a portion of the head of column 121. It also may be desirable, in other cases, to employ the embodiment shown in Figure 5.

**[0043]** As shown in Figure 5, an additional distillation column 541 has been added to the process. This column receives argon-enriched vapour stream 139 as a feed and produces oxygen-depleted stream 545 from the top. Stream 545 is at least partially condensed in heat exchanger 147 to form stream 549, which subsequently is split into two portions, 551 and 553. Stream 553 is ultimately an argon product, but may contain nitrogen and oxygen and, therefore, may be subjected to further purification steps. Stream 551 is returned to column 541 as reflux, flows downward through the column, exits the bottom, is pumped in pump 543 if necessary, then is returned to partitioned section 125 as stream 143. Column 541 may provide a wide range of separation stages. Typically, 20 to 200 stages of separation will be used, depending on the desired oxygen content of stream 545.

**[0044]** Alternatively, one may elect to further purify argon-enriched stream 139 using a means other than distillation. For example, the argon-enriched stream may be removed from the process and passed to an adsorption separation system (not shown) for the removal of oxygen, nitrogen, or both. Such an adsorption separation may take place in a single bed or in multiple beds and may be carried out at cold, warm or even hot temperatures. Oxygen may be removed from the argon-enriched stream via a catalytic oxidation step as well. A membrane separation scheme also could be a suitable substitute for purification by distillation. Combinations of distillation and one of the three above mentioned alternatives may be used in conjunction to further purify argon-enriched stream 139.

**[0045]** In the preceding discussions, the partitioned section 125 received vapour stream 137 as a bottom feed. As shown in Figure 6, it also is possible to configure the partitioned section to receive a liquid as a top feed. The feeds to and from the higher pressure column 103 are the same as for Figures 1 and 5. However, the liquid stream 633 exiting the top distillation section 623 of the lower pressure column 121 is split into two streams: a first portion 635 and a second portion 637. First portion 635 flows into the intermediate distillation section 127. Second portion 637 flows into the partitioned section 125, defined by the vertical separating element 129 and end separating element 131. In this embodiment, the end section 131 is at the bottom of the vertical separating element 129 unlike the embodiments of Figures 1 and 5 (where it is at the top). Liquid stream 637 descends through the partitioned section 125 and is distilled to produce argon-enriched stream 639.

Stream 639 is at least partially vaporized in heat exchanger 147 to produce stream 641, which subsequently is split into two streams, 643 and 645. Stream 643 is returned to the partitioned section 125 as boilup; argon-enriched stream 645 is removed from the distillation system. The heat input for heat exchanger 147 is provided by cooling the crude liquid oxygen stream 115. In this mode of operation, stream 637 is substantially free of oxygen and the partitioned section performs a nitrogen-argon separation.

**[0046]** In Figures 1 and 5, refrigeration for heat exchanger 147 is derived by partially vaporizing the crude liquid oxygen stream 115. Persons skilled in the art will recognize that any liquid stream permitting a suitable temperature driving force in that heat exchanger 147 would be a suitable substitute for the crude liquid oxygen stream. Examples of such streams include a condensed air stream or a liquid nitrogen stream.

**[0047]** In Figures 1, 5 and 6, the oxygen product stream 153 is shown as being withdrawn from the lower pressure column 121 as a vapour. However, the present invention is not limited to such an operation. Persons skilled in the art will recognize that oxygen product stream 153 may be withdrawn from the lower pressure column as a liquid, pumped to a higher pressure, then vaporized and warmed. Gaseous oxygen produced in this manner also may be optionally compressed before being delivered to the end user. This technique is commonly referred to as pumped-LOX. To facilitate the vaporization of the pumped oxygen stream, it is common to compress a suitable gas, cool it, and then condense it by indirect heat exchange with the liquid oxygen. Examples of gases used for this purpose include feed air and nitrogen vapour recycled from the air separation unit. When air is used for this purpose, the condensed high pressure air is used as a feed to the higher pressure column 103, the lower pressure column 121, or both.

**[0048]** Condensed air also may be used in the present invention in a manner analogous to crude liquid oxygen. For example, condensed air may be cooled to provide the heat input for heat exchanger 147 in Figure 6. Likewise, after being cooled and/or suitably reduced in pressure, condensed air may be used to provide refrigeration for heat exchanger 147 in Figures 1 and 5. As with condensed air, any liquid stream may alternatively be withdrawn from the higher pressure column and utilized for heat exchanger 147 in Figures 1, 5 and 6.

**[0049]** In Figure 6, heat input to heat exchanger 147 is provided by cooling crude liquid oxygen. As stated above, other suitably warm fluids may be cooled. In addition, a fluid may be condensed in heat exchanger 147 of Figure 6 to provide heat input. Examples of such fluids include a portion of vapour nitrogen from the higher pressure column or a portion of vapour air.

**[0050]** No reference is made in Figures 1, 5, and 6 to the nature of the mass exchange devices in any of the distillation sections. Persons skilled in the art will recognize that any of sieve trays, bubble-cap trays, valve

trays, random packing, or structured packing, used individually or in combination, are suitable for the application of the present invention.

**[0051]** The embodiments of Figures 1, 5, and 6 illustrate the application of the present invention to in a double-column distillation system. It will be understood by persons skilled in the art that the double-column processes shown in these figures are simplified for clarity. Other feeds to the double column system often exist. For example: 1) a portion of the feed air stream may be expanded for refrigeration and fed to the lower pressure column 121; 2) multiple oxygen products may be withdrawn from the lower pressure column; and 3) an additional nitrogen-enriched stream may be withdrawn from a location above feed stream 119 in the lower pressure column 121 or from the higher pressure column 103.

**[0052]** Although double-column configurations are the most common for the recovery of oxygen and argon from air, the present invention is not limited to such configurations. For example, there exist single-column processes for oxygen recovery from air. Such processes may easily incorporate a partitioned section for producing an argon-enriched stream, and in such an event, the present invention would be applicable.

**[0053]** In Figures 1, 5 and 6, the heat exchanger 147 is shown to exist external to the lower pressure column 121. However, it is possible, and in some instances preferred, to locate the heat exchanger 147 inside the lower pressure column 121.

**[0054]** The present invention may be used to separate a multicomponent feed which comprises more than three components. Examples are shown in Figures 7 and 8 and are described below.

**[0055]** Figure 7 shows an example for the separation of a four-component mixture. Component A is the most volatile; component D is the least volatile; and components B and C are of intermediate volatility. The multicomponent feed 709 is introduced to distillation column 701 having a condenser 702, a reboiler 704, an intermediate distillation section 705, and a partitioned section 703. A stream enriched in the most volatile component A is withdrawn from the top of the column 701 as stream 715. In this example, stream 715 also contains one of the intermediate volatility components, B. Stream 711 enriched in the least volatile component D is withdrawn from the bottom of the column 701. Stream 713 enriched in intermediate volatility component C is produced from the partitioned section 703. A portion of this stream is vaporized in reboiler 706 and returned to the partitioned section as boilup. Stream 715 subsequently is fed to a downstream distillation column 707, which has a condenser 708 and a reboiler 710. Column 707 produces a fluid enriched in component A from the top of the column as stream 719 and a fluid enriched in component B from the bottom of the column as stream 717.

**[0056]** Figure 8 shows another example for the separation of a four-component mixture. Component A is the most volatile; and component D is the least volatile;

components B and C are of intermediate volatility. The multicomponent feed 809 is introduced to distillation column 801 having a condenser 802, a reboiler 804, an intermediate distillation section 805 and partitioned section 803. A stream enriched in the most volatile component A is withdrawn from the top of column 801 as stream 815. A stream enriched in the least volatile component D is withdrawn from the bottom of the column 801 as stream 811. In this example, stream 811 also contains one of the intermediate volatility components, C. Stream 813 enriched in intermediate volatility component B is produced from the partitioned section 803. A portion of this stream is condensed in condenser 806 and returned to the partitioned section 803 as reflux. Stream 811 subsequently is fed to a downstream distillation column 807, which has a condenser 808 and a reboiler 810. Column 807 produces a fluid enriched in component C from the top of the column as stream 819 and a fluid enriched in component D from the bottom of the column as stream 817.

**[0057]** It will be apparent to persons skilled in the art that the configurations shown in Figures 7 and 8 also can be applied to feed streams containing more than four components.

**[0058]** Although illustrated and described herein with reference to certain specific embodiments, the present invention is nevertheless not intended to be limited to the precise details shown. Rather, various modifications may be made without departing from the scope of the invention as defined in the following claims.

## Claims

1. A process for distillation of a multicomponent fluid containing at least three components, each component having a different volatility, into at least three streams, using a distillation column system comprising a first distillation column having at least a first distillation section, a second distillation section, an intermediate distillation section between the first distillation section and the second distillation section, and a partitioned section adjacent the intermediate distillation section, which is isolated from the intermediate distillation section by a vertical separating element, and is closed at one end by an end separating element and has an equivalent diameter ( $D_e$ ) at least 60% of the ideal diameter ( $D_i$ ) of the partitioned section, wherein a first portion of a fluid stream in the first distillation column flows into the intermediate distillation section and a second portion of said fluid stream flows into the partitioned section; and a side stream, enriched in a component having an intermediate volatility, is withdrawn from the partitioned section.
2. A process as claimed in Claim 1, wherein the end separating element is at the top of the partitioned

- section and the fluid stream is a vapour rising from a distillation section below the intermediate distillation section.
3. A process as claimed in Claim 2, wherein a liquid is fed to the partitioned section at a location adjacent the top of the partitioned section.
  4. A process as claimed in Claim 3, wherein the liquid is produced by at least partially condensing at least a portion of a vapour leaving the partitioned section.
  5. A process as claimed in any one of Claims 2 to 4, wherein the partitioned section extends upwardly beyond the level of the intermediate section.
  6. A process as claimed in Claim 1, wherein the end separating element is at the bottom of the partitioned section and the fluid stream is a liquid descending from a distillation section above the intermediate distillation section.
  7. A process as claimed in Claim 6, wherein a vapour is fed to the partitioned section at a location adjacent the bottom of the partitioned section.
  8. A process as claimed in Claim 7, wherein the vapour is produced by at least partially vaporizing a portion of the liquid leaving the partitioned section.
  9. A process as claimed in any one of Claims 6 to 8, wherein the partitioned section extends downwardly below the level of the intermediate section.
  10. A process as claimed in any one of the preceding claims, wherein the vertical separating element is cylindrical.
  11. A process as claimed in any one of Claims 1 to 10, wherein the vertical separating element comprises a vertical wall attached to a cylindrical wall of the first distillation column.
  12. A process as claimed in any one of the preceding claims, wherein the side stream is transferred to at least one other distillation column.
  13. A process as claimed in any one of the preceding claims, wherein a stream enriched in a component having the highest volatility is withdrawn from a location above at least one distillation section above the intermediate distillation section; and a stream enriched in a component having the lowest volatility is withdrawn from a location below at least one distillation section below the intermediate distillation section.
  14. A process as claimed in any one of the preceding claims in which the separation is cryogenic.
  15. A process as claimed in Claim 14, wherein the multicomponent fluid mixture is a nitrogen/oxygen/argon mixture.
  16. A process as claimed in Claim 15, wherein the multicomponent fluid is air.
  17. A process as claimed in Claim 16, wherein the argon-enriched stream withdrawn from the partitioned section has an oxygen content of less than 60 mole%.
  18. A process as claimed in Claim 16 or Claim 17, wherein the argon-enriched stream withdrawn from the partitioned section is transferred to at least one other distillation column.
  19. A process as claimed in any one of Claims 1 to 13, wherein the multicomponent fluid is selected from benzene/toluene/xylene mixtures, nitrogen/carbon monoxide/methane mixtures, combinations of three or more components from C<sub>1</sub> to C<sub>5</sub> alcohols, and hydrocarbon mixtures, said hydrocarbon mixtures being selected from the group consisting of pentane-hexane-heptane, isopentane-pentane-hexane, butane-isopentane-pentane, iso-butane-n-butane-gasoline, and combinations of three or more components from C<sub>1</sub> to C<sub>6</sub> hydrocarbons or C<sub>4</sub> isomers.
  20. A distillation column for use in a process of Claim 1, said distillation column (121) having a first distillation section (123); a second distillation section; an intermediate distillation section (127) between the first distillation section and the second distillation section; and a partitioned section (125) adjacent the intermediate distillation section (127) isolated from the intermediate distillation section (127) by a vertical separating element (129) and closed at one end by an end separating element (131) and having an equivalent diameter (De) at least 60% of the ideal diameter (Di) of the partitioned section.
  21. A distillation column system as claimed in Claim 20, wherein the vertical separating element (129) is cylindrical.
  22. A distillation column system as claimed in Claim 20, wherein the vertical separating element (129) comprises a vertical wall attached to a cylindrical wall of the distillation column.
  23. A distillation column system for separation of a multicomponent fluid containing at least three components by a process as defined in Claim 1, said system comprising:



a distillation column (121) as defined in Claim 20;  
conduit means (119) for feeding the multicomponent fluid to the distillation column (121);  
conduit means (151) for withdrawing from the top of the column (121) a stream enriched in a component having the highest volatility;  
conduit means (153) for withdrawing from the bottom of the column (121) a stream enriched in a component having the lowest volatility; and  
conduit means (139) for withdrawing from the partitioned section a side stream enriched in a component having an intermediate volatility.

24. A system as claimed in Claim 23 which is adapted to conduct a process as defined in any one of Claims 2 to 18.

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FIG. 1

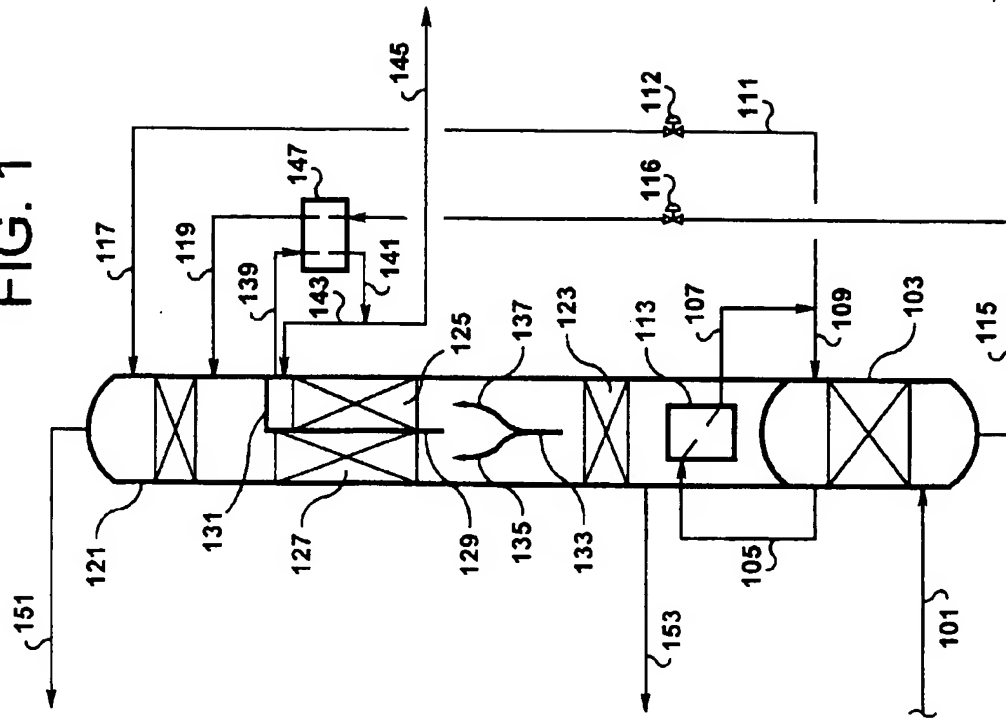


FIG. 2B

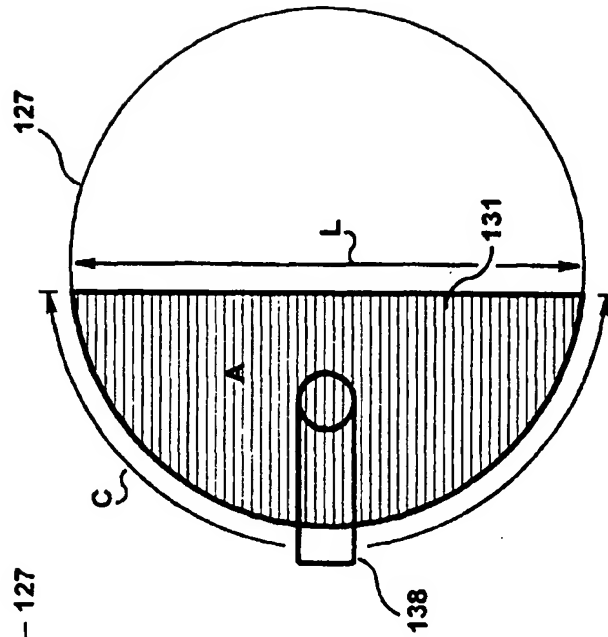
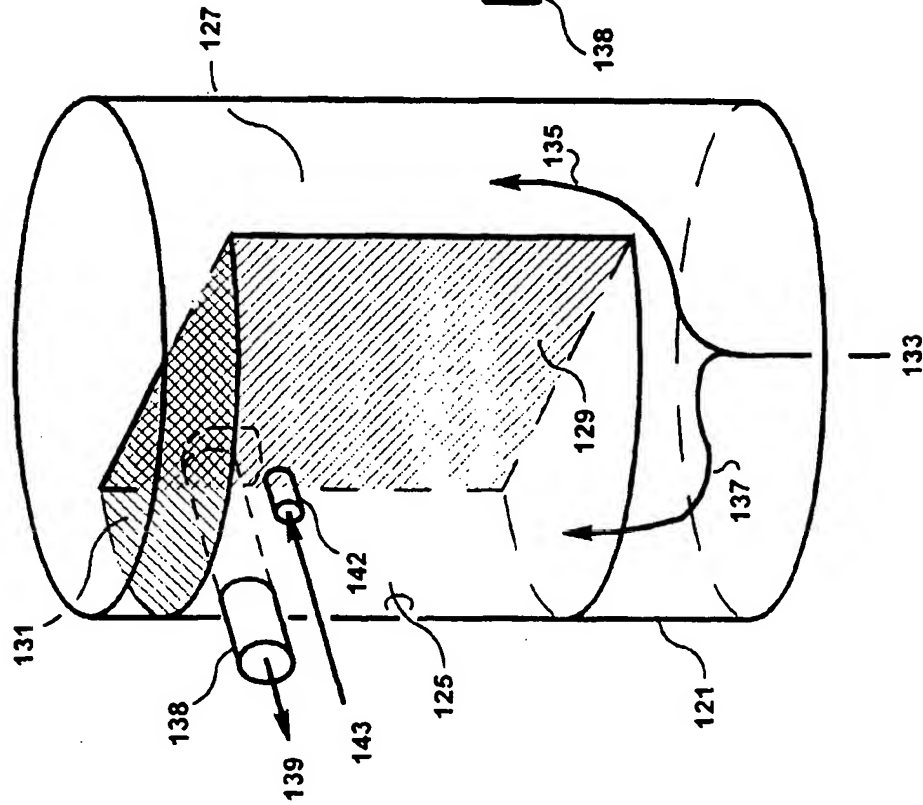
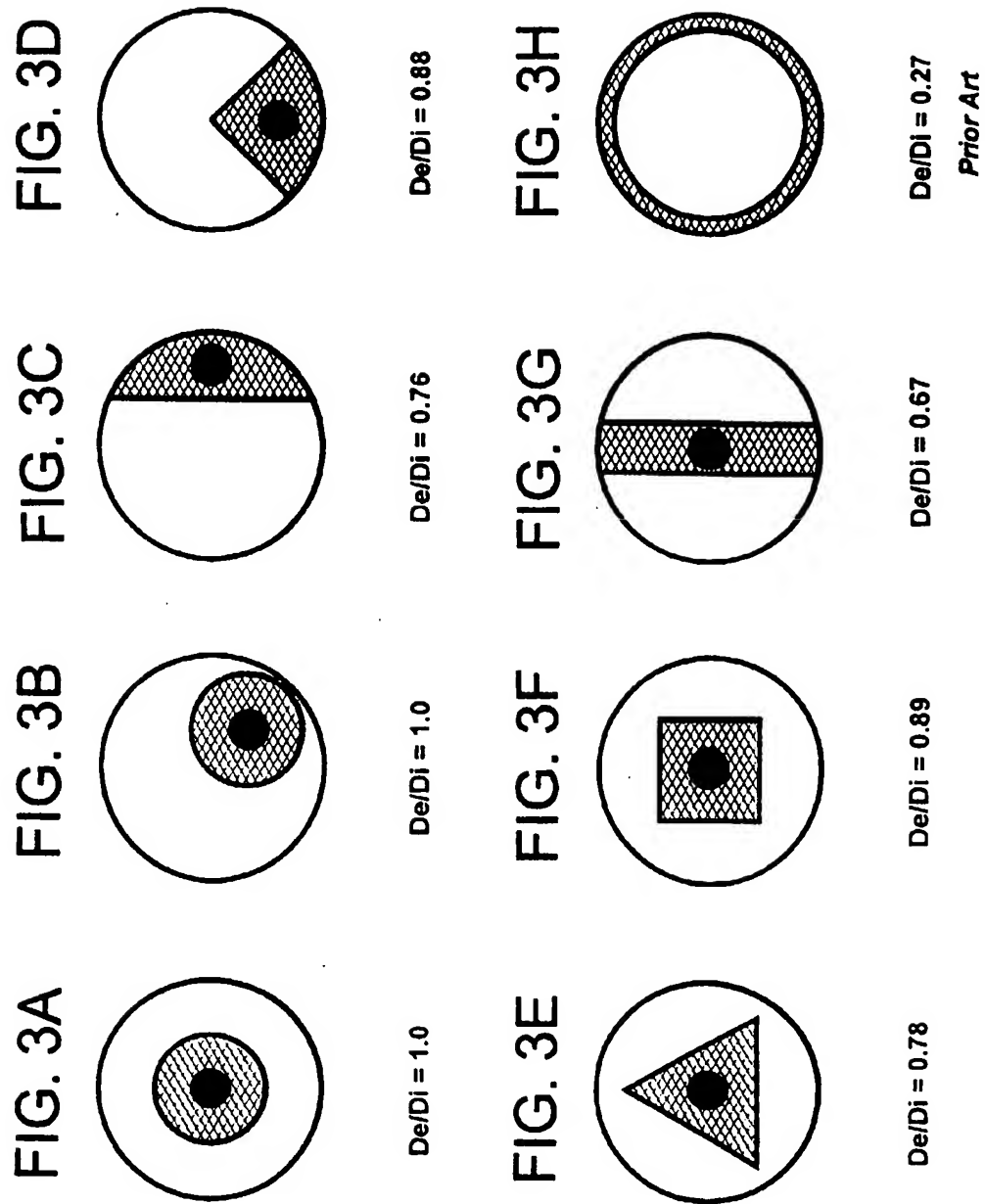


FIG. 2A





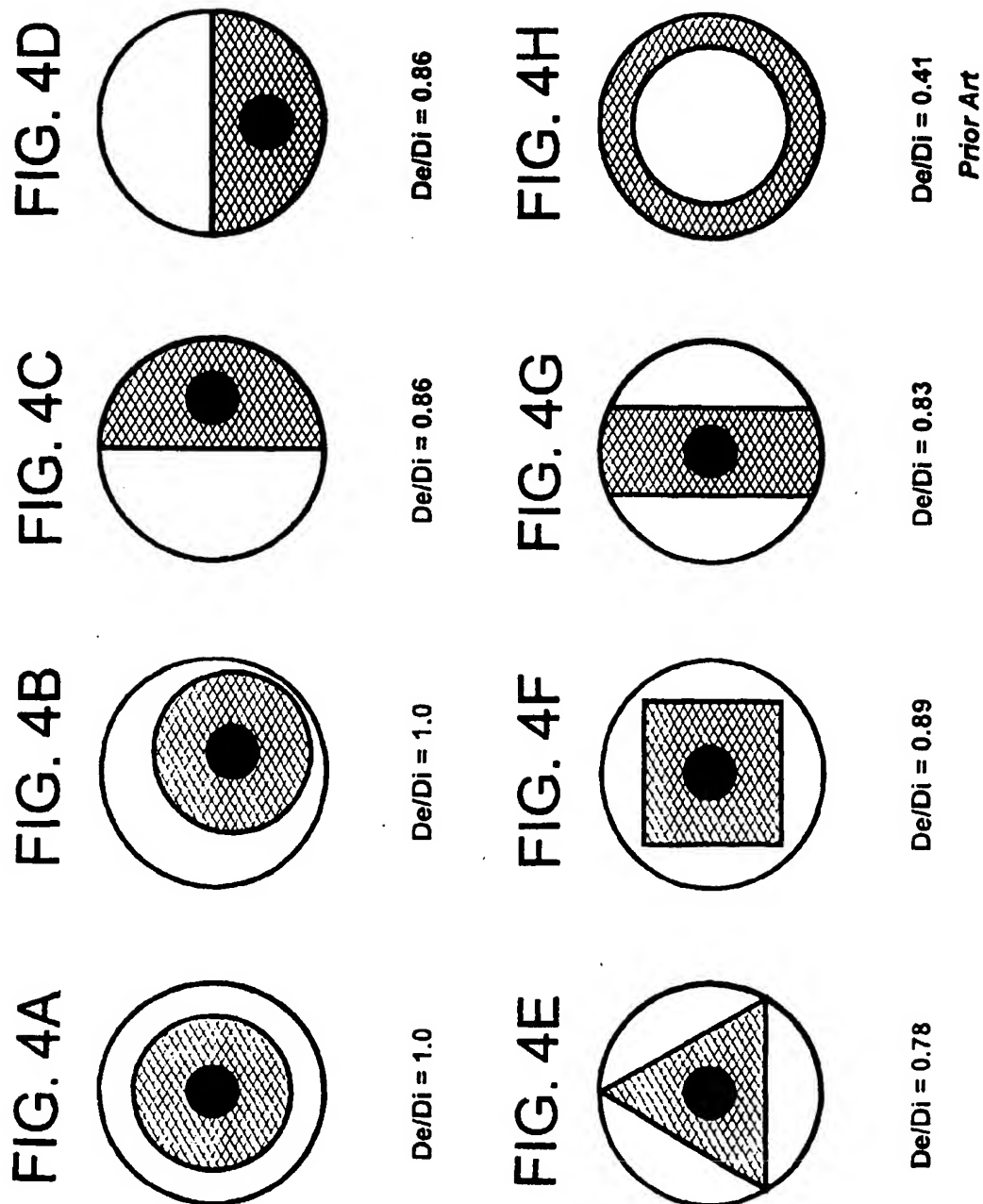


FIG. 5

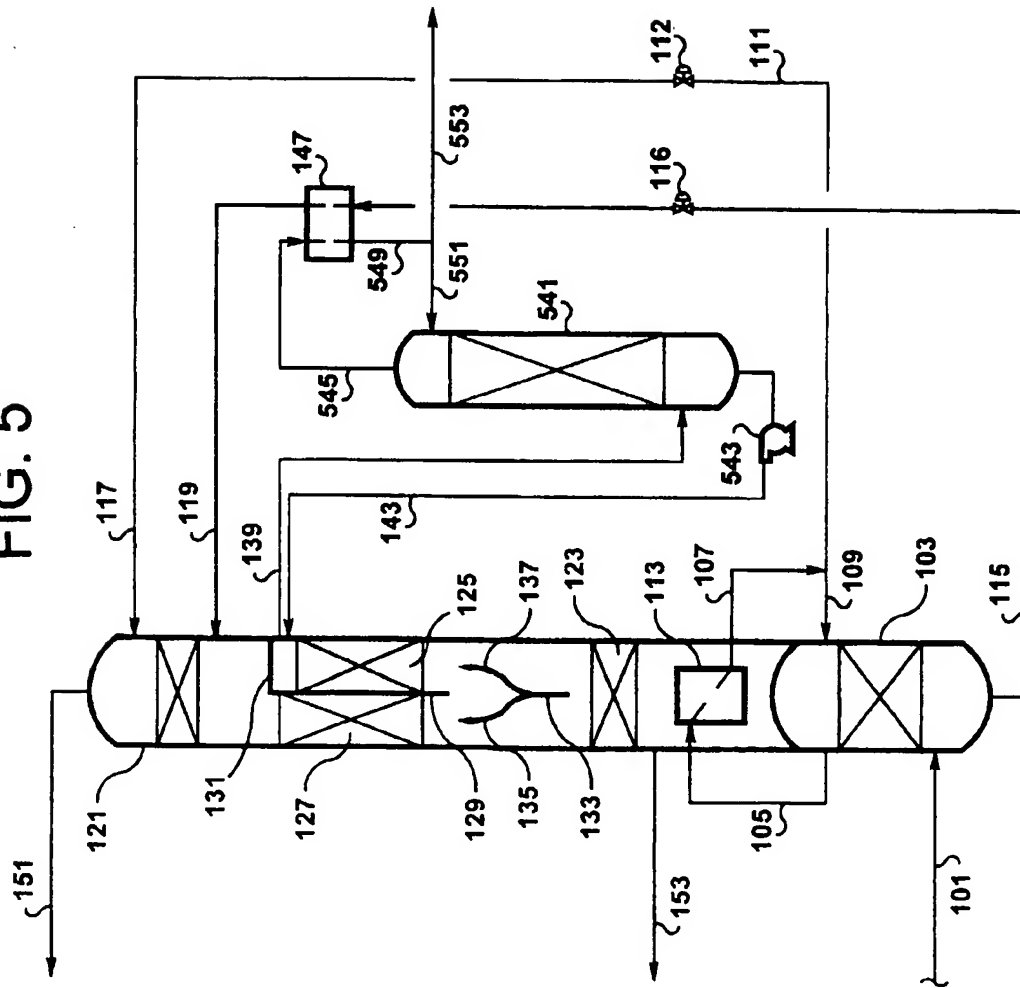


FIG. 6

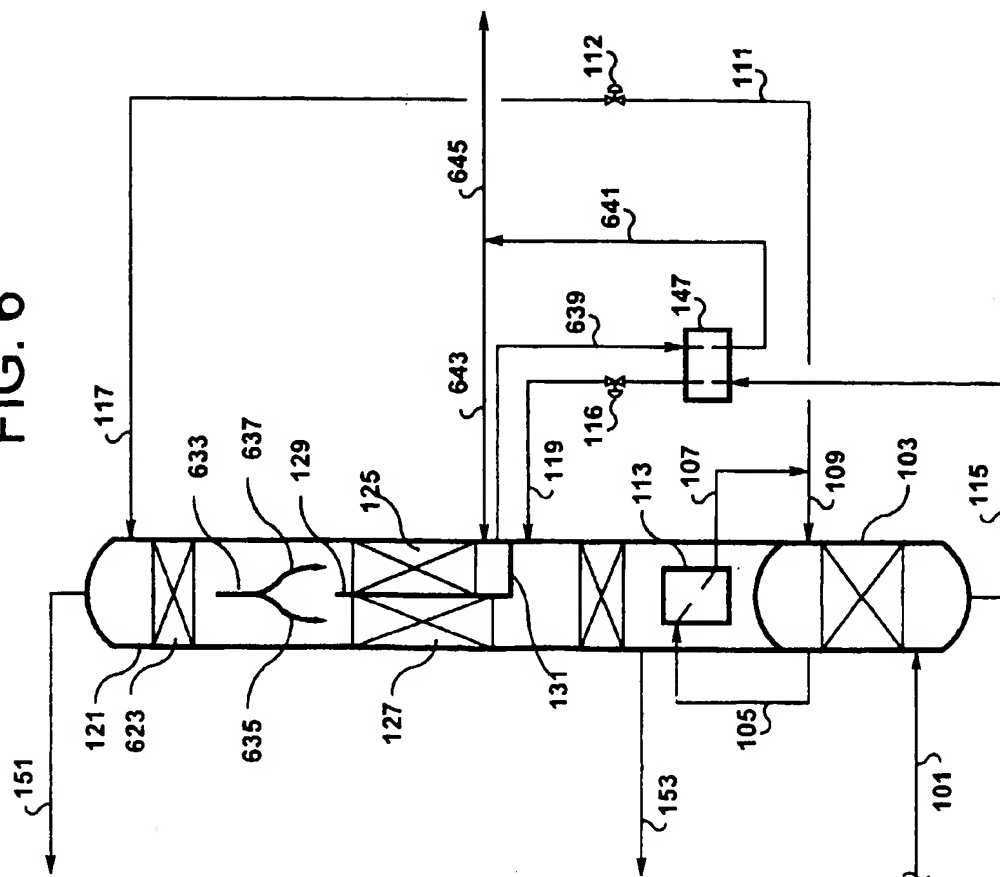


FIG. 7

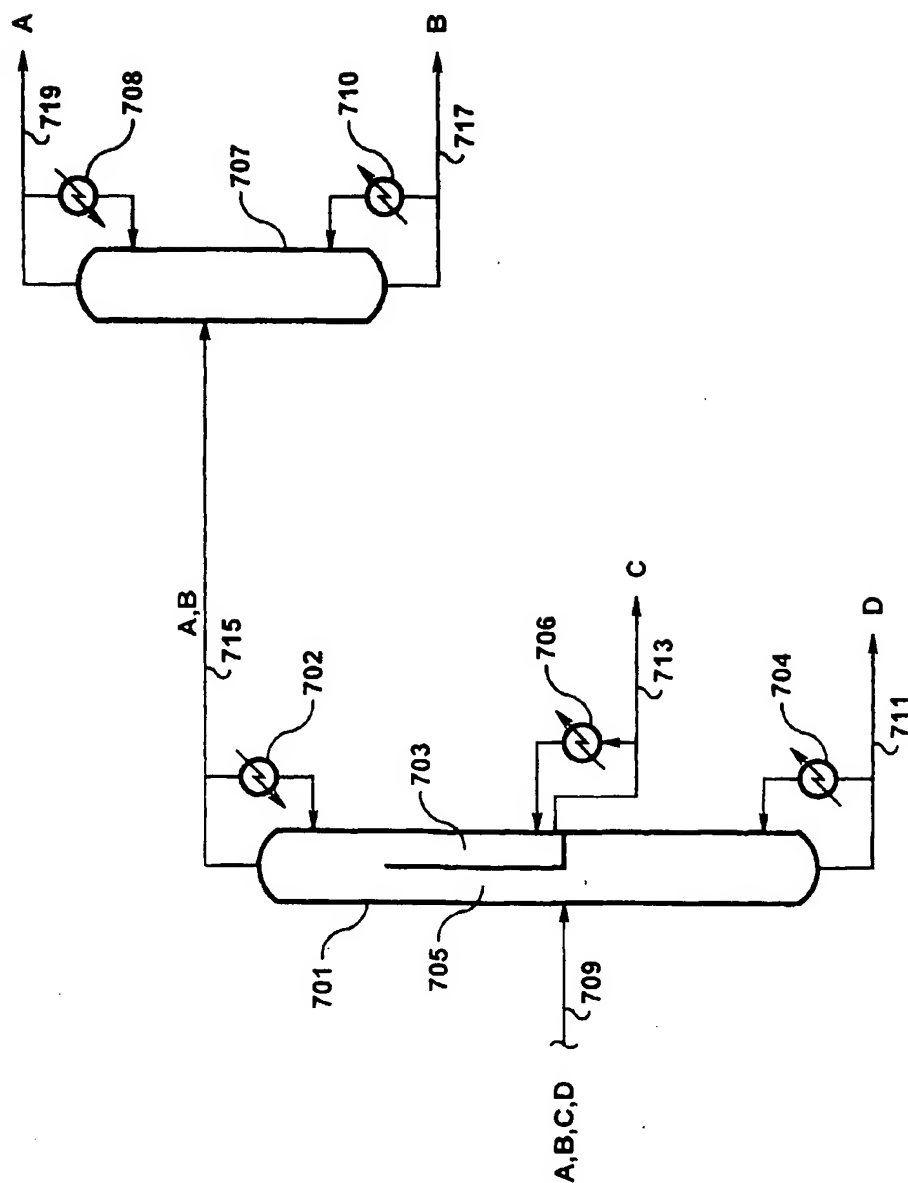
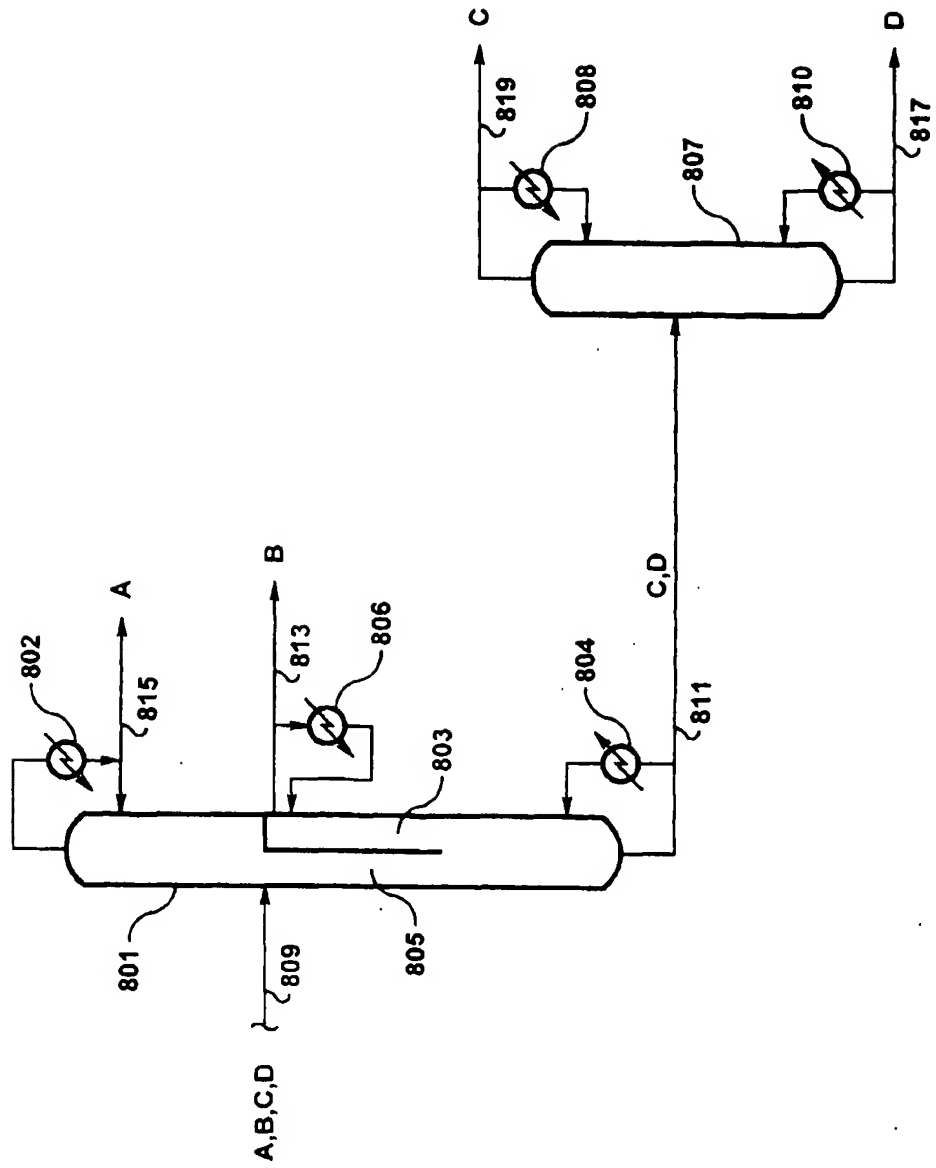




FIG. 8





European Patent  
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## EUROPEAN SEARCH REPORT

Application Number

EP 00 31 0946

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Place of search <b>THE HAGUE</b>		Date of completion of the search <b>8 March 2001</b>	Examiner <b>Lapeyrere, J</b>
<div>CATEGORY OF CITED DOCUMENTS</div> <div>           X: particularly relevant if taken alone            Y: particularly relevant if combined with another document of the same category            A: technological background            O: non-written disclosure            P: intermediate document         </div> <div>           T: theory or principle underlying the invention            E: earlier patent document, but published on, or after the filing date            D: document cited in the application            L: document cited for other reasons            &amp;: member of the same patent family, corresponding document         </div>			

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